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(54) CENTRIFUGAL SEPARATION OF BISPHENOL A-PHENOL ADDUCT

(57)Abstract:

PURPOSE: To carry out a long-term stable operation by preventing vibration of a filtration tank caused by, e.g. concentration change in centrifugal separation of crystallized bisphenol A-phenol adduct from a phenol solution (slurry) of bisphenol A.

CONSTITUTION: This invention relates to a method for supplying slurry to the bottom of a filtration tank and forming a cake through a cake formation zone. In this method, the formation of a cake is carried out while making a uniform cake formation zone by continuously breaking an abnormally grown cake in a cake formation zone using a cake formation control liquid.

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CLAIMS

[Claim(s)]

[Claim 1] a slurry to this addition product with which the phenol addition product of bisphenol A is contained by carrying out crystallization in the phenol solution of bisphenol A -- a centrifugal-filtration separator -- a cake -- pass a generation zone -- the approach of carrying out filtration separation as a cake -- setting -- the cake of a lauter-tub internal surface -- some cakes [at least] currently generated in a generation zone -- a cake -- the centrifugal-filtration separation approach of the phenol addition product of bisphenol A characterized by to carry out filtration separation, running off with the liquid for runoff.

[Claim 2] a cake -- the centrifugal filtration separation approach of the phenol addition product of bisphenol A according to claim 1 that the liquid for runoff is a phenol content solution.

[Claim 3] a cake -- the centrifugal filtration separation approach of the phenol addition product of bisphenol A according to claim 1 that the liquid for runoff is a phenol solution of bisphenol A by which filtration separation was carried out.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the approach of carrying out centrifugal filtration separation of the phenol addition product of bisphenol A. It is related more with the approach a long duration deer also carries out centrifugal filtration separation of the phenol addition product of bisphenol A stably from the phenol solution of bisphenol A at a detail.

[0002]

[Description of the Prior Art] Although bisphenol A equivalent to 2 and 2-screw (4-hydroxyl phenyl) propane is widely used as main raw materials, such as an epoxy resin and polycarbonate resin, for the quality improvement demanded in the particular application of these resin, bisphenol A of a high grade is needed. As one of the particular application of this, the need as an optical material grows in polycarbonate resin in recent years, and supply of bisphenol A of a high grade is demanded by colorlessness more than before.

[0003] As the purification approach of this bisphenol A in the process which manufactures bisphenol A industrially, bisphenol A builds a phenol and an addition product and there is a method of using the property which carries out crystallization into a phenol solution. That is, if bisphenol A is manufactured by using a phenol and an acetone as the main raw material, the phenol solution of bisphenol A which contains the crystal and manufacture impurity of a phenol addition product of bisphenol A by crystallization actuation will be obtained by the slurry regime.

[0004] The centrifugal filtration separator besides a belt filter, a tray filter, and a drum filter has been conventionally used for this solid liquid separation. In the case of the belt filter which is a suction type among filtration-type solid-liquid-separation machines various [these], a tray filter, and a drum filter, since many adhesion liquid is contained in the solid-state layer after solid liquid separation, it has the fault of causing the increment in the amount of evaporation phenols at the time of dissolving the phenol addition product of bisphenol A at a back process, and distilling off a phenol.

[0005] On the other hand, since a centrifugal filtration separator uses the centrifugal force like [it cannot measure gravity] with it and equips the inside of a plane with the lauter tub which has a side attachment wall for solid-liquid filtration separation, it can dissociate almost completely quickly [a solid-liquid-separation rate], and, on the other hand, it is a separator which can also use recovery of a liquid component conveniently if it is easy and an object is not the thing of a colloidal state. [large] In fact, in order to separate the crystal of the phenol addition product of bisphenol A from the phenol solution (slurry) of bisphenol A with which crystallization actuation was performed, the above-mentioned function of this centrifugal filtration separator is suitable, and use application was often tried.

[0006] However, although the problem of the increment in an evaporation load with adhesion liquid like [in the case of the above-mentioned suction type] is avoided when this centrifugal-filtration separator is used, the phenomenon of unusual oscillating generating of a lauter tub was often seen, the remarkable fall of the solid-liquid-separation engine performance took place, and destabilization of the centrifugal-filtration condition by which this oscillating generating is accompanied led also to the variation in the quality of bisphenol A which is a final product, and

has been made into a problem. The trouble of this oscillating generating is analyzed in more detail, and is seen. In order to often operate a centrifugal filtration separator Although stable maintenance of the revolving shaft of an internal lauter tub is the most important When separating the crystal of the above-mentioned addition product, this addition product sets to a lauter tub wall at the circumferential direction. To an ununiformity Namely, it will be easy to be in the condition that it deposits asymmetrically to a medial axis in many cases, therefore the support condition of a lauter tub cannot serve as imbalance, the unusual vibration cannot be caused, and the stable revolving shaft cannot be maintained for a long time. Consequently, it is easy to cause the unusual contact to the body of a centrifugal filtration separator, and the rotating lauter tub, decline in a filtration efficiency, promotion of uneven deposition of this addition product to a tub wall, exfoliation of this addition product from a tub internal surface, etc.

[0007] Although it is not certain about the device in which the special phenomenon of uneven deposition generating in crystal separation of the phenol addition product of this bisphenol A happens, it is thought by generating of this phenomenon itself that selection of slurry concentration or the concentration change under that supply has influenced considerably. For example, when taking a continual process, it is usually the phenol solution of 0% of said addition product concentration at the time of a start up. Although concentration change is not avoided in order to take the service condition which raises slurry concentration gradually and goes, it is easy to generate at the time of this concentration change and the concentration change based on fluctuation of the crystallization operating condition added to the phenol solution of bisphenol A etc., Although slurry concentration is usually about 20 - 40 % of the weight from points, such as a fluidity at the time of supply, and supply efficiency It becomes unstable even if the concentration is fixed, if rotation of a lauter tub will be stable if fixed at such within the limits, and it becomes low concentration from 20 % of the weight considerably, Moreover, it is observed that the supply condition of a slurry will become unstable if it exceeds 40 % of the weight far conversely, and rotation becomes unstable as a result. In addition, it complicates a process and is not desirable, although the proposal which establishes the slurry concentration process of a cyclone is also in the preceding paragraph of a centrifugal filtration separation process in order to make it not supply the above low-concentration slurries. As mentioned above, although the case where said addition product begins to carry out uneven deposition was described, if ununiformity-ization once takes place and a lauter tub begins to vibrate, a call and the phenomenon in which vibration becomes intense will also be further seen in ununiformity-ization.

[0008]

[Problem(s) to be Solved by the Invention] Therefore, the thing for which generating of vibration of the lauter tub in the process to which solid liquid separation of the slurry which consists of a phenol addition product of bisphenol A of the phenol solution and crystal of bisphenol A is carried out using a centrifugal filtration separator is prevented in case the technical problem of this invention manufactures bisphenol A, Moreover, although it is in extinguishing this even if it generates, finally it can carry out by solid-liquid-separation operation carrying out long duration stability by this vibration isolation, and is in quality at fewer things of variation for which it enables it to produce bisphenol A of high quality more.

[0009]

[Means for Solving the Problem] [when the crystal which consists of a phenol addition product of bisphenol A is filtered as a result of repeating research wholeheartedly, in order that this invention persons may solve the above-mentioned technical problem, and it is going to deposit as a cake on a lauter tub wall inside the plane gradually from the slurry regime] A phenol solution is sprayed on an uneven deposition part. if deposition of this crystal begins to occur in an ununiformity at the circumferential direction in a lauter tub -- immediately -- this -- When passing away and changing into the deposition condition of a symmetrical cake as much as possible to the uniform condition, i.e., a medial axis, at the circumferential direction, it came to complete a header and this invention for rotation being stabilized. namely, the slurry contained by the phenol addition product of bisphenol A carrying out crystallization of the summary of this

invention into the phenol solution of bisphenol A to this addition product -- a centrifugal-filtration separator -- a cake -- pass a generation zone -- the approach of carrying out filtration separation as a cake -- setting -- the cake of a lauter-tub internal surface -- some cakes [at least] which are generating in a generation zone -- a cake -- it is in the centrifugal-filtration separation approach of the phenol addition product of bisphenol A which carries out filtration separation, running off with the liquid (a cake generation control liquid) for runoff.

[0010] Hereafter, the contents of this invention are explained to a detail. Generally the centrifugal filtration separator used in this invention is not used for the application, and is not limited especially. That is, they are the lauter tub which rotates one shaft inside structurally at high speed, and the centrifugal filtration separator with which the body discharged in response to filtrate was arranged outside. Since a slurry is supplied continuously and filtration separation is immediately carried out in a lauter tub internal surface in order to perform centrifugal filtration separation by the continual process, a revolving shaft is not limited to a vertical type or a water flat tip. However, maintenance of rotational stability is more easy for a vertical type, and a water flat tip has the description, respectively at the point that the ejection of an addition product by which filtration separation was carried out is easy.

[0011] The path of the filtration hole of a lauter tub internal surface is suitably chosen in consideration of factors, such as particle size distribution of the above-mentioned phenol addition product, and hardness of an addition product (crystal). Since a centrifugal force is applied and the surface tension of filtrate is hardly satisfactory, it is as much as possible detailed and more desirable to prepare much punching in respect of separation efficiency. A body receives the filtrate which passed the above-mentioned filtration wall, if there is a function which discharges, is enough and can use the same thing as the thing of the usual centrifugal filtration separator.

[0012] Although a slurry may be supplied to the filtering area of a lauter tub wall with various means in order to perform centrifugal filtration separation Jet supply of the slurry is once carried out at the filtration bottom of the tank section and the pars basilaris ossis occipitalis which formed the distributor preferably. The approach of pushing aside this slurry to homogeneity at a surrounding lauter tub internal-surface side, and moving in the direction of lauter tub opening along with this lauter tub internal surface further The homogeneity of the slurry supply to a circumferencial direction, the plasticity of the uniform slurry concentration inclination (inclination of filtration enrichment) in the direction of a revolving shaft, the slurry under filtration which is in a lauter tub internal surface with a slurry supply pressure, or the cake after filtration -- a deposit can move to a lauter tub opening side -- further -- the cake after this filtration -- a deposit is desirable in respect of being easy to take out from this opening continuously etc. in addition -- although various paths of this distributor can be chosen, if it brings close to a lauter tub bore -- said cake -- the function which extrudes a deposit besides a tub at a piston type can be given, and it is convenient.

[0013] Although a slurry should just prepare a supply nozzle in the filtration bottom of the tank section movable, from the purpose which raises the homogeneity of the slurry supply in a filtration bottom of the tank section circumferencial direction, a book may be arranged circularly and it may install them. [many] Punching is suitably prepared along this annular section periphery edge of the slurry supply pipe which furthermore has the annular section in a filtration container, and the method which makes a slurry blow off from this punching can also be used conveniently.

[0014] If the concentration of the phenol solution of the phenol addition product of bisphenol A used in the invention in this application is 20 - 40 % of the weight generally made suitable in centrifugal filtration separation technology conventionally [said], it is the most suitable, but if the approach of the invention in this application is used, it is applicable even zero to at least 50% of the weight. That is, since according to the approach of the invention in this application the removal correction of the ununiformity section is continuously made so that it may mention later, it is usable conventionally also in the low concentration or the high concentration field from which it was easy to start oscillating generating of a lauter tub. In addition, although the higher one of the concentration of bisphenol A is desirable when adjusting the above-mentioned

addition product concentration to 0 - 50% of the weight (preferably 20 - 40 % of the weight), what is necessary is just 0 - 50 % of the weight. Moreover, although slurry temperature influences slurry viscosity and it is related to filtration velocity, generally 20-80 degrees C is suitable also including handling nature.

[0015] In making a lauter tub internal surface carry out the fine-particles deposition of the phenol addition product of bisphenol A as a cake which does not almost have a fluidity using the above-mentioned centrifugal filtration separator according to the approach of the invention in this application this -- the cake in the condition that there is still a fluidity before a cake is generated by homogeneity at a circumferencial direction -- some cakes [at least] generated in the generation zone -- a cake, sprinkling the liquid (cake generation control liquid) which can run off, and making it run off suitably It is going to make a circumferencial direction generate the deposit of a cake to homogeneity. When the generation process of this cake is observed (for example, observation by the speed light photography synchronized with the filtration container engine speed) and the centrifugal filtration separator is working normally while a slurry moves to a lauter tub opening side -- a uniform liquefied object to a short time -- ***** of a configuration -- a uniform cake -- becoming -- therefore -- yet -- a fluidity -- it is -- a cake with an unstable configuration, since the die length of a generation zone becomes very short this cake -- ***** a flow unusual in a generation zone occurs for a while and cake-izes a part -- this -- a cake -- the heterogeneity of the circumferencial direction of a generation zone is considered that there is no effect in destabilization of rotation of a lauter tub.

[0016] however -- if slurry concentration falls or there is fluctuation -- this cake -- the generation zone became long, and it was observed so that the variation in this die length in a circumferencial direction might become size. therefore -- yet -- a fluidity -- it is -- a cake with an unstable configuration -- fluctuation of the die length of a generation zone -- immediately -- the part of a cake -- influencing -- this -- thing ununiformity-ization in the die length of the circumferencial direction of the part of a cake and thickness occurs, and vibration of a lauter tub will begin to take place to coincidence -- it comes out. if this abnormality more specifically begins to occur -- this cake -- the inside of a generation zone -- a cake -- the shape of an island -- moreover -- a circumferencial direction -- an ununiformity -- being distributed -- generating -- between these islands -- yet -- a part -- the condition that the slurry was flowing was able to see. the slurry which flows in the direction of lauter tub opening through between islands probably because a rate also goes up a flow between narrow islands -- a cake ---izing is also behind. it -- contrary -- an island -- colliding -- the late slurry of flow -- a cake ---izing was also quick, and it was observed as it was increasingly made greatly and thick, the island, i.e., the cake, already made. It is thought that these vicious circles are calling the imbalance of rotation of a lauter tub.

[0017] the proposal by this invention persons was made based on this observation result, and is the above-mentioned sheep -- a dovetail shape-like cake with an unstable fluidity -- it is going to break down from the outside the cake which was generated in the generation zone and which is usually an island-like by spraying of another liquid (cake generation control liquid). the part of the cake generated in the shape of [this] an island -- from the method of the inside of a lauter tub -- this -- a cake, when sprinkling the liquid for runoff an island-like cake -- breaking down -- again -- fluidization, i.e., a cake,, since it is the purpose to return to the same condition as a generation zone a cake -- although the liquid for runoff is usually good at spraying of spraying extent from a nozzle, since it will not collapse simply if the case where an island-like cake is large, and filtration are progressing, spraying by the strong jet from a nozzle is also needed. Moreover, if distance of the location at the tip of a nozzle and a lauter tub wall is made adjustable, since it can respond to the condition of the island cake of the above, it is convenient. Furthermore, since neither die length nor a location is fixed, if two or more steps of nozzles are installed in the direction of a revolving shaft of a lauter tub, the cake of the shape of this island can break down an un-uniformity cake quickly over the large range, and is effective. Furthermore, although it is not limited, if said purpose is taken into consideration, it is desirable [especially the liquid spraying direction from a nozzle / sprinkling to radial / of a lauter tub] also in respect of effectiveness also in respect of few quantification [**** / making it

concentrate only on a part [the pars insularis] of a spray.

[0018] the cake sprinkled from the above-mentioned nozzle -- since an island-like cake must be broken down first, the liquid for runoff will not be limited especially if it is the liquid which can achieve this purpose. however, the cake after breaking down as mentioned above -- since it is also important to return to the same condition as a generation zone, and to make it a uniform cake again, the mother liquor which uses the liquid containing the phenol which constitutes a slurry, for example, water, a phenolated water solution, the bisphenol A content phenol solution, and the filtered bisphenol A content phenol solution as a principal component is suitable also in respect of effectiveness also in respect of effectiveness again.

[0019] such a cake -- spraying of the liquid for runoff (cake generation control liquid) -- an island-like cake -- a cake -- although it returns to the condition of a generation zone, since a part for the insular part by which filtration enrichment was carried out is again made into the shape of a slurry -- a surrounding original cake -- there is no difference not much with the concentration of the part of a generation zone. therefore, these -- both -- a cake -- filtration progresses to coincidence, and both generation zones become a cake and go. In addition, a cake is generated in the direction which increases thickness again, and goes in the direction which opens the cake already generated, and, on the other hand, an island-like cake disappearing continuously and going is observed.

[0020] Hereafter, an example explains the invention in this application.

[Example]

(Example 1) Centrifugal filtration separation actuation was performed in rotational frequency 1000rpm using the centrifugal filtration separator 1 of the horizontal type which builds in the bore of 800mm shown in drawing 1, 1600mm of revolving-shaft length, and the lauter tub 2 of 150l. of content volume. It has distributor 2a, and rotates, and movable ejector-plate 2b is prepared in the revolving-shaft 3 direction, and supply pipe 4b of a slurry 4 which has opening 4a near this distributor is inserted in the pars basilaris ossis occipitalis of a lauter tub 2 from the lauter tub opening side. moreover, the cake which carries out opening toward a lauter tub internal surface -- the nozzles 5a, 5b, and 5c for the liquid 5 (cake generation control liquid) jet for runoff are formed. In addition, bulb 5f for flow regulation v and 5m of flowmeters are attached in these nozzles, respectively. The crystallization can (not shown) to which crystallization of bisphenol A is carried out from the phenol solution of bisphenol A was filled with the phenolated water solution which is a solvent at first, this 70-degree C phenolated water solution was charged from this crystallization can in 32t /to the lauter tub in an hour, and idling was started. All the phenolated water solutions that are feed liquid were filtered and collected. Vibration of the lauter tub under this charge and operation of filtration was 0.02cm/second in rate conversion. Although supply of bisphenol A was started with the crystallization can and it waited for the rise of the bisphenol A concentration of a crystallization can, and the slurry concentration supplied to a lauter tub, when slurry concentration became about 7% of the weight, the oscillating value of a lauter tub rose rapidly and became a second in 0.26cm /. the place which observed the internal surface of a lauter tub at this time -- a circumferencial direction -- an ununiformity -- an island-like cake -- the layer was generating. The slurry with the other liquefied part was flowing. then, the maximum upstream edge of an island-like cake layer -- a cake -- the generation control liquid nozzles 5a and 5b -- since it did -- a cake -- bulb 5v of generation control liquid nozzle 5a -- opening -- a cake -- flow control was tried in order to equalize a layer. a cake -- the cake of the shape of an island of the range on which generation control liquid 5 is sprayed -- a layer -- disappearing -- a cake -- a cake uniform among the generation control liquid nozzles 5a and 5b -- the layer generated. the cake at this time -- the amount of generation control liquid flows was 3t/hour. in addition, a cake -- the collected mother liquor (bisphenol A content phenol solution) was used for generation control liquid. Vibration fell [second] in 0.05cm /by rate conversion. the place which observed the interior of a lauter tub again when slurry concentration went up and it became 30% of the weight of a convention -- a cake -- a cake more uniform upstream than the spraying range from generation control liquid nozzle 5a -- since the layer was generating -- a cake -- generation control liquid bulb 5v was stopped.

[0021] (Example 2) The same centrifugal filtration separator as an example 1 and the crystallization can were used, and supply of the phenol to a crystallization can, charge to a lauter tub, and idling were performed on an example 1 and these conditions. Vibration of the lauter tub at the time was 0.02cm/second in rate conversion like the case of an example 1. Supply of bisphenol A was started with the crystallization can, and it waited for the rise of the bisphenol A concentration of a crystallization can, and the supply slurry concentration to a lauter tub. When slurry concentration was 5 % of the weight, the oscillating value of a lauter tub rose rapidly and became a second in 0.28cm /. the place which observed the interior of a lauter tub at this time -- a circumferencial direction -- an ununiformity -- an island-like cake -- the layer was generating. The slurry with the other liquefied part was flowing. the maximum upstream edge of the cake layer of the shape of an uneven island -- a cake -- since the generation control liquid nozzles 5a and 5b did -- a cake -- bulb 5v of generation control liquid nozzle 5a -- opening -- a cake -- flow control was tried in order to equalize a layer. a cake -- the cake of the shape of an island of the range on which generation control liquid 5 is sprayed -- although the layer disappeared -- a down-stream cake -- between the generation control liquid nozzles 5b and 5c -- again -- an island-like cake -- the layer generated. the cake at this time -- the amount of generation control liquid flows was 4t/hour. in addition, a cake -- the collected mother liquor was used for generation control liquid. Vibration was 0.29cm/second in rate conversion. a cake -- equalization of the island cake layer of the above generated among the generation control liquid nozzles 5b and 5c -- it should carry out -- a cake -- bulb 5v of generation control liquid nozzle 5b was opened, and flow control was tried. The bulb of the above-mentioned upstream was also adjusted. two cakes -- the cake of the shape of an island of the range which generation control liquid has sprayed -- a layer -- disappearing -- a uniform cake -- the layer generated. the cake at this time -- the amount of generation control liquid flows was [2t /and 5c of 5b] 3t/hour an hour. a cake -- generation control liquid used the collected mother liquor. Vibration fell [second] in 0.09cm /by rate conversion. the place which observed the interior of a lauter tub again since slurry concentration went up and it became 30% of the weight of a convention -- a cake -- a cake more uniform upstream than the spraying range from generation control liquid nozzle 5a -- since the layer was generating -- both cakes -- generation control liquid bulb 5v was stopped.

[0022] (Example 3) The same centrifugal filtration separator as an example 1 and the crystallization can were used, and supply of the phenol to a crystallization can, charge to a lauter tub, and idling were performed on an example 1 and these conditions. Vibration of the lauter tub at the time was 0.02cm/second in rate conversion like the case of an example 1. Supply of bisphenol A was started with the crystallization can, and it waited for the rise of the bisphenol A concentration of a crystallization can, and the supply slurry concentration to a lauter tub. When slurry concentration was 9 % of the weight, the oscillating value of a lauter tub rose rapidly and became a second in 0.22cm /. the place which observed the interior of a lauter tub at this time -- a circumferencial direction -- an ununiformity -- an island-like cake -- the layer was generating. The slurry with the other liquefied part was flowing. the maximum upstream edge of the cake layer of the shape of an uneven island -- a cake -- since generation control liquid nozzle 5aa and 5bb(s) did -- a cake -- bulb 5v of generation control liquid nozzle 5a -- opening -- a cake -- flow control was tried in order to equalize a layer. a cake -- the cake of the shape of an island of the range on which generation control liquid 5 is sprayed -- a layer -- disappearing -- a cake -- a cake uniform among the generation control liquid nozzles 5a and 5b -- the layer generated. the cake at this time -- the amount of generation control liquid flows was 4t/hour. in addition, a cake -- the collected mother liquor was used for generation control liquid. Vibration fell [second] in 0.06cm /by rate conversion. the place which observed the interior of a lauter tub again since slurry concentration went up and it became 15% of the weight of a convention -- a cake -- the cake more uniform than the spraying range from generation control liquid nozzle 5a on a lower stream of a river -- since the layer was generating, operation was continued as it was.

[0023]

[Effect of the Invention] the time of carrying out solid liquid separation of the slurry which

consists of a phenol solution of bisphenol A, and a crystallized phenol addition product of bisphenol A using a centrifugal-filtration separator -- vibration of a lauter tub -- the cake of a lauter-tub wall -- even if preventing vibration of a lauter tub by filtering the cake unusually generated in the generation zone, running off with a phenol solution etc. and vibration occur, this can extinguish, and it can carry out now by being stabilized solid-liquid-separation operation for a long time.

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TECHNICAL FIELD

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[0004] The centrifugal filtration separator besides a belt filter, a tray filter, and a drum filter has been conventionally used for this solid liquid separation. In the case of the belt filter which is a suction type among filtration-type solid-liquid-separation machines various [these], a tray filter, and a drum filter, since many adhesion liquid is contained in the solid-state layer after solid liquid separation, it has the fault of causing the increment in the amount of evaporation phenols at the time of dissolving the phenol addition product of bisphenol A at a back process, and distilling off a phenol.

[0005] On the other hand, since a centrifugal filtration separator uses the centrifugal force like [it cannot measure gravity] with it and equips the inside of a plane with the lauter tub which has a side attachment wall for solid-liquid filtration separation, it can dissociate almost completely quickly [a solid-liquid-separation rate], and, on the other hand, it is a separator which can also use recovery of a liquid component conveniently if it is easy and an object is not the thing of a colloidal state. [large] In fact, in order to separate the crystal of the phenol addition product of bisphenol A from the phenol solution (slurry) of bisphenol A with which crystallization actuation was performed, the above-mentioned function of this centrifugal filtration separator is suitable, and use application was often tried.

[0006] However, although the problem of the increment in an evaporation load with adhesion liquid like [in the case of the above-mentioned suction type] is avoided when this centrifugal-filtration separator is used, the phenomenon of unusual oscillating generating of a lauter tub was often seen, the remarkable fall of the solid-liquid-separation engine performance took place, and destabilization of the centrifugal-filtration condition by which this oscillating generating is accompanied led also to the variation in the quality of bisphenol A which is a final product, and has been made into a problem. The trouble of this oscillating generating is analyzed in more detail, and is seen. In order to often operate a centrifugal filtration separator, It will be easy to be in the condition that the revolving shaft which this addition product accumulated [in / although stability maintenance of the revolving shaft of an internal lauter tub is most important, when separating the crystal of the above-mentioned addition product / the circumferential direction] on the lauter tub wall asymmetrically to the ununiformity, i.e., a medial axis, in many cases, therefore the support condition of a lauter tub became imbalance, caused the unusual vibration,

and was stabilized is unmaintainable for a long time. Consequently, it is easy to cause the unusual contact to the body of a centrifugal filtration separator, and the rotating lauter tub, decline in a filtration efficiency, promotion of uneven deposition of this addition product to a tub wall, exfoliation of this addition product from a tub internal surface, etc.

[0007] Although it is not certain about the device in which the special phenomenon of uneven deposition generating in crystal separation of the phenol addition product of this bisphenol A happens, it is thought by generating of this phenomenon itself that selection of slurry concentration or the concentration change under that supply has influenced considerably. When taking a continual process, in order to usually take the service condition of 0% of said addition product concentration which is a phenol solution, raises slurry concentration gradually and goes at the time of a start up Although it being easy to generate at the time of this concentration change and the concentration change based on fluctuation of the crystallization operating condition added to the phenol solution of bisphenol A etc. although concentration change is not avoided, and slurry concentration are usually about 20 - 40 % of the weight from points, such as a fluidity at the time of supply, and supply efficiency It becomes unstable even if the concentration is fixed, if rotation of a lauter tub will be stable if fixed at such within the limits, and it becomes low concentration from 20 % of the weight considerably, Moreover, it is observed that the supply condition of a slurry will become unstable if it exceeds 40 % of the weight far conversely, and rotation becomes unstable as a result. In addition, it complicates a process and is not desirable, although the proposal which establishes the slurry concentration process of a cyclone is also in the preceding paragraph of a centrifugal filtration separation process in order to make it not supply the above low-concentration slurries. As mentioned above, although the case where said addition product begins to carry out uneven deposition was described, if ununiformity-ization once takes place and a lauter tub begins to vibrate, a call and the phenomenon in which vibration becomes intense will also be further seen in ununiformity-ization.

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EFFECT OF THE INVENTION

[Effect of the Invention] the time of carrying out solid liquid separation of the slurry which consists of a phenol solution of bisphenol A, and a crystallized phenol addition product of bisphenol A using a centrifugal-filtration separator -- vibration of a lauter tub -- the cake of a lauter-tub wall -- even if preventing vibration of a lauter tub by filtering the cake unusually generated in the generation zone, running off with a phenol solution etc. and vibration occur, this can extinguish, and it can carry out now by being stabilized solid-liquid-separation operation for a long time.

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] Therefore, the thing for which generating of vibration of the lauter tub in the process to which solid liquid separation of the slurry which consists of a phenol addition product of bisphenol A of the phenol solution and crystal of bisphenol A is carried out using a centrifugal filtration separator is prevented in case the technical problem of this invention manufactures bisphenol A. Moreover, although it is in extinguishing this even if it generates, finally it can carry out by solid-liquid-separation operation carrying out long duration stability by this vibration isolation, and is in quality at fewer things of variation for which it enables it to produce bisphenol A of high quality more.

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MEANS

[Means for Solving the Problem] [when the crystal which consists of a phenol addition product of bisphenol A is filtered as a result of repeating research wholeheartedly, in order that this invention persons may solve the above-mentioned technical problem, and it is going to deposit as a cake on a lauter tub wall inside the plane gradually from the slurry regime] A phenol solution is sprayed on an uneven deposition part. if deposition of this crystal begins to occur in an ununiformity at the circumferencial direction in a lauter tub -- immediately -- this -- When passing away and changing into the deposition condition of a symmetrical cake as much as possible to the uniform condition, i.e., a medial axis, at the circumferencial direction, it came to complete a header and this invention for rotation being stabilized. namely, the slurry contained by the phenol addition product of bisphenol A carrying out crystallization of the summary of this invention into the phenol solution of bisphenol A to this addition product -- a centrifugal-filtration separator -- a cake -- pass a generation zone -- the approach of carrying out filtration separation as a cake -- setting -- the cake of a lauter-tub internal surface -- some cakes [at least] which are generating in a generation zone -- a cake -- it is in the centrifugal-filtration separation approach of the phenol addition product of bisphenol A which carries out filtration separation, running off with the liquid (a cake generation control liquid) for runoff.

[0010] Hereafter, the contents of this invention are explained to a detail. Generally the centrifugal filtration separator used in this invention is not used for the application, and is not limited especially. That is, they are the lauter tub which rotates one shaft inside structurally at high speed, and the centrifugal filtration separator with which the body discharged in response to filtrate was arranged outside. Since a slurry is supplied continuously and filtration separation is immediately carried out in a lauter tub internal surface in order to perform centrifugal filtration separation by the continual process, a revolving shaft is not limited to a vertical type or a water flat tip. However, maintenance of rotational stability is more easy for a vertical type, and a water flat tip has the description, respectively at the point that the ejection of an addition product by which filtration separation was carried out is easy.

[0011] The path of the filtration hole of a lauter tub internal surface is suitably chosen in consideration of factors, such as particle size distribution of the above-mentioned phenol addition product, and hardness of an addition product (crystal). Since a centrifugal force is applied and the surface tension of filtrate is hardly satisfactory, it is as much as possible detailed and more desirable to prepare much punching in respect of separation efficiency. A body receives the filtrate which passed the above-mentioned filtration wall, if there is a function which discharges, is enough and can use the same thing as the thing of the usual centrifugal filtration separator.

[0012] Although a slurry may be supplied to the filtering area of a lauter tub wall with various means in order to perform centrifugal filtration separation Jet supply of the slurry is once carried out at the filtration bottom of the tank section and the pars basilaris ossis occipitalis which formed the distributor preferably. The approach of pushing aside this slurry to homogeneity at a surrounding lauter tub internal-surface side, and moving in the direction of lauter tub opening along with this lauter tub internal surface further The homogeneity of the slurry supply to a circumferencial direction, the plasticity of the uniform slurry concentration inclination (inclination

of filtration enrichment) in the direction of a revolving shaft, the slurry under filtration which is in a lauter tub internal surface with a slurry supply pressure, or the cake after filtration -- a deposit can move to a lauter tub opening side -- further -- the cake after this filtration -- a deposit is desirable in respect of being easy to take out from this opening continuously etc. in addition -- although various paths of this distributor can be chosen, if it brings close to a lauter tub bore -- said cake -- the function which extrudes a deposit besides a tub at a piston type can be given, and it is convenient.

[0013] Although a slurry should just prepare a supply nozzle in the filtration bottom of the tank section movable, from the purpose which raises the homogeneity of the slurry supply in a filtration bottom of the tank section circumferencial direction, a book may be arranged circularly and it may install them. [many] Punching is suitably prepared along this annular section periphery edge of the slurry supply pipe which furthermore has the annular section in a filtration container, and the method which makes a slurry blow off from this punching can also be used conveniently.

[0014] If the concentration of the phenol solution of the phenol addition product of bisphenol A used in the invention in this application is 20 - 40 % of the weight generally made suitable in centrifugal filtration separation technology conventionally [said], it is the most suitable, but if the approach of the invention in this application is used, it is applicable even zero to at least 50% of the weight. That is, since according to the approach of the invention in this application the removal correction of the ununiformity section is continuously made so that it may mention later, it is usable conventionally also in the low concentration or the high concentration field from which it was easy to start oscillating generating of a lauter tub. In addition, although the higher one of the concentration of bisphenol A is desirable when adjusting the above-mentioned addition product concentration to 0 - 50% of the weight (preferably 20 - 40 % of the weight), what is necessary is just 0 - 50 % of the weight. Moreover, although slurry temperature influences slurry viscosity and it is related to filtration velocity, generally 20-80 degrees C is suitable also including handling nature.

[0015] In making a lauter tub internal surface carry out the fine-particles deposition of the phenol addition product of bisphenol A as a cake which does not almost have a fluidity using the above-mentioned centrifugal filtration separator according to the approach of the invention in this application this -- the cake in the condition that there is still a fluidity before a cake is generated by homogeneity at a circumferencial direction -- some cakes [at least] generated in the generation zone -- a cake, sprinkling the liquid (cake generation control liquid) which can run off, and making it run off suitably It is going to make a circumferencial direction generate the deposit of a cake to homogeneity. When the generation process of this cake is observed (for example, observation by the speed light photography synchronized with the filtration container engine speed) and the centrifugal filtration separator is working normally while a slurry moves to a lauter tub opening side -- a uniform liquefied object to a short time -- ***** of a configuration -- a uniform cake -- becoming -- therefore -- yet -- a fluidity -- it is -- a cake with an unstable configuration, since the die length of a generation zone becomes very short this cake -- ***** a flow unusual in a generation zone occurs for a while and cake-izes a part -- this -- a cake -- the heterogeneity of the circumferencial direction of a generation zone is considered that there is no effect in destabilization of rotation of a lauter tub.

[0016] however -- if slurry concentration falls or there is fluctuation -- this cake -- the generation zone became long, and it was observed so that the variation in this die length in a circumferencial direction might become size. therefore -- yet -- a fluidity -- it is -- a cake with an unstable configuration -- fluctuation of the die length of a generation zone -- immediately -- the part of a cake -- influencing -- this -- thing ununiformity-ization in the die length of the circumferencial direction of the part of a cake and thickness occurs, and vibration of a lauter tub will begin to take place to coincidence -- it comes out. if this abnormality more specifically begins to occur -- this cake -- the inside of a generation zone -- a cake -- the shape of an island -- moreover -- a circumferencial direction -- an ununiformity -- being distributed -- generating -- between these islands -- yet -- a part -- the condition that the slurry was flowing was able to see. the slurry which flows in the direction of lauter tub opening through between

islands probably because a cake also goes up a flow between narrow islands -- a cake ---izing is also behind. it -- contrary -- an island -- colliding -- the late slurry of flow -- a cake ---izing was also quick, and it was observed as it was increasingly made greatly and thick, the island, i.e., the cake, already made. It is thought that these vicious circles are calling the imbalance of rotation of a lauter tub.

[0017] the proposal by this invention persons was made based on this observation result, and is the above-mentioned sheep -- a dovetail shape-like cake with an unstable fluidity -- it is going to break down from the outside the cake which was generated in the generation zone and which is usually an island-like by spraying of another liquid (cake generation control liquid). the part of the cake generated in the shape of [this] an island -- from the method of the inside of a lauter tub -- this -- a cake, when sprinkling the liquid for runoff an island-like cake -- breaking down -- again -- fluidization, i.e., a cake,, since it is the purpose to return to the same condition as a generation zone a cake -- although the liquid for runoff is usually good at spraying of spraying extent from a nozzle, since it will not collapse simply if the case where an island-like cake is large, and filtration are progressing, spraying by the strong jet from a nozzle is also needed. Moreover, if distance of the location at the tip of a nozzle and a lauter tub wall is made adjustable, since it can respond to the condition of the island cake of the above, it is convenient. Furthermore, since neither the length nor a location is fixed, if two or more steps of nozzles are installed in the direction of a revolving shaft of a lauter tub, the cake of the shape of this island can break down an un-uniformity cake quickly over the large range, and is effective. Furthermore, although it is not limited, if said purpose is taken into consideration, it is desirable [especially the liquid spraying direction from a nozzle / sprinkling to radial / of a lauter tub] also in respect of effectiveness also in respect of few quantification [**** / making it concentrate only on a part for the pars insularis] of a spray.

[0018] the cake sprinkled from the above-mentioned nozzle -- since an island-like cake must be broken down first, the liquid for runoff will not be limited especially if it is the liquid which can achieve this purpose. however, the cake after breaking down as mentioned above -- since it is also important to return to the same condition as a generation zone, and to make it a uniform cake again, the mother liquor which uses the liquid containing the phenol which constitutes a slurry, for example, water, a phenolated water solution, the bisphenol A content phenol solution, and the filtered bisphenol A content phenol solution as a principal component is suitable also in respect of effectiveness also in respect of effectiveness again.

[0019] such a cake -- spraying of the liquid for runoff (cake generation control liquid) -- an island-like cake -- a cake -- although it returns to the condition of a generation zone, since a part for the insular part by which filtration enrichment was carried out is again made into the shape of a slurry -- a surrounding original cake -- there is no difference not much with the concentration of the part of a generation zone. therefore, these -- both -- a cake -- filtration progresses to coincidence, and both generation zones become a cake and go. In addition, a cake is generated in the direction which increases thickness again, and goes in the direction which opens the cake already generated, and, on the other hand, an island-like cake disappearing continuously and going is observed.

[0020] Hereafter, an example explains the invention in this application.

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EXAMPLE

[Example]

(Example 1) Centrifugal filtration separation actuation was performed in rotational frequency 1000rpm using the centrifugal filtration separator 1 of the horizontal type which builds in the bore of 800mm shown in drawing 1, 1600mm of revolving-shaft length, and the lauter tub 2 of 150l. of content volume. It has distributor 2a, and rotates, and movable ejector-plate 2b is prepared in the revolving-shaft 3 direction, and supply pipe 4b of a slurry 4 which has opening 4a near this distributor is inserted in the pars basilaris ossis occipitalis of a lauter tub 2 from the lauter tub opening side. moreover, the cake which carries out opening toward a lauter tub internal surface -- the nozzles 5a, 5b, and 5c for the liquid 5 (cake generation control liquid) jet for runoff are formed. In addition, bulb 5f for flow regulation v and 5m of flowmeters are attached in these nozzles, respectively. The crystallization can (not shown) to which crystallization of bisphenol A is carried out from the phenol solution of bisphenol A was filled with the phenolated water solution which is a solvent at first, this 70-degree C phenolated water solution was charged from this crystallization can in 32t /to the lauter tub in an hour, and idling was started. All the phenolated water solutions that are feed liquid were filtered and collected. Vibration of the lauter tub under this charge and operation of filtration was 0.02cm/second in rate conversion. Although supply of bisphenol A was started with the crystallization can and it waited for the rise of the bisphenol A concentration of a crystallization can, and the slurry concentration supplied to a lauter tub, when slurry concentration became about 7% of the weight, the oscillating value of a lauter tub rose rapidly and became a second in 0.26cm /. the place which observed the internal surface of a lauter tub at this time -- a circumferential direction -- an ununiformity -- an island-like cake -- the layer was generating. The slurry with the other liquefied part was flowing, then, the maximum upstream edge of an island-like cake layer -- a cake -- the generation control liquid nozzles 5a and 5b -- since it did -- a cake -- bulb 5v of generation control liquid nozzle 5a -- opening -- a cake -- flow control was tried in order to equalize a layer. a cake -- the cake of the shape of an island of the range on which generation control liquid 5 is sprayed -- a layer -- disappearing -- a cake -- a cake uniform among the generation control liquid nozzles 5a and 5b -- the layer generated. the cake at this time -- the amount of generation control liquid flows was 3t/hour. in addition, a cake -- the collected mother liquor (bisphenol A content phenol solution) was used for generation control liquid. Vibration fell [second] in 0.05cm /by rate conversion. the place which observed the interior of a lauter tub again when slurry concentration went up and it became 30% of the weight of a convention -- a cake -- a cake more uniform upstream than the spraying range from generation control liquid nozzle 5a -- since the layer was generating -- a cake -- generation control liquid bulb 5v was stopped.

[0021] (Example 2) The same centrifugal filtration separator as an example 1 and the crystallization can were used, and supply of the phenol to a crystallization can, charge to a lauter tub, and idling were performed on an example 1 and these conditions. Vibration of the lauter tub at the time was 0.02cm/second in rate conversion like the case of an example 1. Supply of bisphenol A was started with the crystallization can, and it waited for the rise of the bisphenol A concentration of a crystallization can, and the supply slurry concentration to a lauter tub. When

slurry concentration was 5 % of the weight, the oscillating value of a lauter tub rose rapidly and became a second in 0.28cm /. the place which observed the interior of a lauter tub at this time -- a circumferencial direction -- an ununiformity -- an island-like cake -- the layer was generating. The slurry with the other liquefied part was flowing. the maximum upstream edge of the cake layer of the shape of an uneven island -- a cake -- since the generation control liquid nozzles 5a and 5b did -- a cake -- bulb 5v of generation control liquid nozzle 5a -- opening -- a cake -- flow control was tried in order to equalize a layer. a cake -- the cake of the shape of an island of the range on which generation control liquid 5 is sprayed -- although the layer disappeared -- a down-stream cake -- between the generation control liquid nozzles 5b and 5c -- again -- an island-like cake -- the layer generated. the cake at this time -- the amount of generation control liquid flows was 4t/hour. in addition, a cake -- the collected mother liquor was used for generation control liquid. Vibration was 0.29cm/second in rate conversion. a cake -- equalization of the island cake layer of the above generated among the generation control liquid nozzles 5b and 5c -- it should carry out -- a cake -- bulb 5v of generation control liquid nozzle 5b was opened, and flow control was tried. The bulb of the above-mentioned upstream was also adjusted. two cakes -- the cake of the shape of an island of the range which generation control liquid has sprayed -- a layer -- disappearing -- a uniform cake -- the layer generated. the cake at this time -- the amount of generation control liquid flows was [2t /and 5c of 5b] 3t/hour an hour. a cake -- generation control liquid used the collected mother liquor. Vibration fell [second] in 0.09cm /by rate conversion. the place which observed the interior of a lauter tub again since slurry concentration went up and it became 30% of the weight of a convention -- a cake -- a cake more uniform upstream than the spraying range from generation control liquid nozzle 5a -- since the layer was generating -- both cakes -- generation control liquid bulb 5v was stopped.

[0022] (Example 3) The same centrifugal filtration separator as an example 1 and the crystallization can were used, and supply of the phenol to a crystallization can, charge to a lauter tub, and idling were performed on an example 1 and these conditions. Vibration of the lauter tub at the time was 0.02cm/second in rate conversion like the case of an example 1. Supply of bisphenol A was started with the crystallization can, and it waited for the rise of the bisphenol A concentration of a crystallization can, and the supply slurry concentration to a lauter tub. When slurry concentration was 9 % of the weight, the oscillating value of a lauter tub rose rapidly and became a second in 0.22cm /. the place which observed the interior of a lauter tub at this time -- a circumferencial direction -- an ununiformity -- an island-like cake -- the layer was generating. The slurry with the other liquefied part was flowing. the maximum upstream edge of the cake layer of the shape of an uneven island -- a cake -- since generation control liquid nozzle 5aa and 5bb(s) did -- a cake -- bulb 5v of generation control liquid nozzle 5a -- opening -- a cake -- flow control was tried in order to equalize a layer. a cake -- the cake of the shape of an island of the range on which generation control liquid 5 is sprayed -- a layer -- disappearing -- a cake -- a cake uniform among the generation control liquid nozzles 5a and 5b -- the layer generated. the cake at this time -- the amount of generation control liquid flows was 4t/hour. in addition, a cake -- the collected mother liquor was used for generation control liquid. Vibration fell [second] in 0.06cm /by rate conversion. the place which observed the interior of a lauter tub again since slurry concentration went up and it became 15% of the weight of a convention -- a cake -- the cake more uniform than the spraying range from generation control liquid nozzle 5a on a lower stream of a river -- since the layer was generating, operation was continued as it was.

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] It is the conceptual diagram of one example of the equipment used in order to enforce the approach concerning the invention in this application.

[Description of Notations]

1: Centrifugal filtration separator

2: Lauter tub

3: Revolving shaft

4: Slurry

5: a cake -- the liquid for runoff (cake generation control liquid)

5a, 5b, and 5c: -- a cake -- the nozzle for generation control liquid jet

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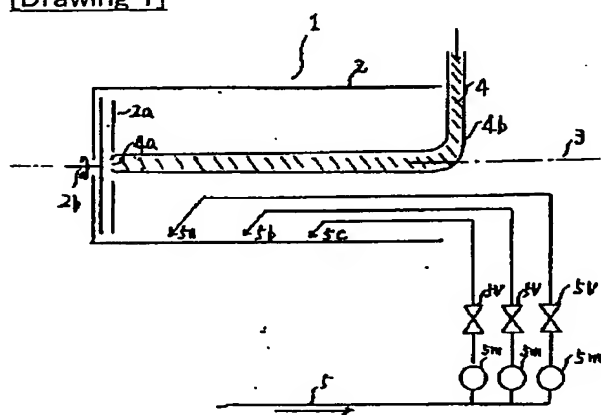
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DRAWINGS

[Drawing 1]



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